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FEASIBILITY STUDY
OF COLUMBIUM ALLOY CASTINGS

JANUARY 1970

G. LANE
Avco Lycoming Division
Stratford, Connecticut

FIRST QUARTERLY PROGRESS REPORT
COVERING 1 AUGUST THROUGH 31 OCTOBER 1969

CONTRACT DAAG46-69-C-0163

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Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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FEASIBILITY STUDY OF COLUMBIUM ALLOY CASTINGS

Technical Report by

G. LANE
Avco Lycoming Division
Stratford, Connecticut

JANUARY 1970

MANUFACTURING METHODS AND TECHNOLOGY PROJECT

First Quarterly Progress Report

Contract DAAG46-69-C-0163

AMCAMS Code 4097.92.9 P6013(X69)

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ABSTRACT

This is the first quarterly report of a program to determine the feasibility of investment casting aluminum alloys presently under investigation. Consumable electrodes of the alloys are arc melted into a water-cooled copper crucible (skull casting) to produce a molten pool of the metal. The liquid metal is subsequently poured into tungsten interfaced shell molds. These molds are produced by the REMET process in which initial tungsten slurry dips provide the non-reactive refractory surface. Thirty-four molds are scheduled in the program to optimize shell design, mold preheat temperatures and, finally, to produce usable castings for the properties testing phase. Configurations include the fluidity pattern, stress rupture bars, oxidation-thermal fatigue paddles, and Lycoming first stage T55 turbine nozzle vanes. Columbium alloys WC-3015 and B-66 were selected for study.

Practice pours of columbium alloy CB-752 and program pours of alloys B-66 and WC-3015 indicate that these materials can be melted and cast into threaded test-bar shapes. Surface quality and soundness of these preliminary castings were acceptable. Surface contamination was not detectable for two of the alloys and only slightly so for the third.

FOREWORD

This technical report is the first quarterly progress report submitted in compliance with Army Contract DAAG46-69-C-0163, and covers the scope of work accomplished during the period of 1 August through 31 October 1969. Proposal 2190.6.1 implements the materials manufacturing project authorized by the contract.

The research and development task dealt with in this report was initiated between the Army Materials and Mechanics Research Center and Avco Lycoming Division to study the feasibility of producing columbium alloy parts by investment casting techniques, and is related to the overall Materials Manufacturing Technology Program.

G. F. Lane, Development Engineer, is project engineer of this study. B. A. Ewing, Chief of Metals and Coating Subsection, has overall responsibility for project management. The study program is under the direction of W. R. Freeman, Jr., Director of the Avco Lycoming Materials Laboratories. This contract is technically supervised by K. D. Holmes of the Army Materials and Mechanics Research Center.

This project has been accomplished as part of the U. S. Army Manufacturing and Technology Program, which has as its objective the timely establishment of manufacturing processes, techniques or equipment to insure the efficient production of current or future defense programs.

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I. INTRODUCTION

In a gas turbine engine one of the most effective means of increasing overall engine performance is to increase the turbine inlet temperature. The limiting factor in the adoption of higher turbine inlet temperatures has been the high temperature materials capability of the hot-end components. At the present time, cobalt and nickel-base alloys in both wrought and cast forms are being extensively used in such applications. Although protective aluminide coatings have extended the high temperature capabilities of these alloys, their low creep rupture strength and the microstructural instability of the required coating systems generally limit their usefulness above 1800°F.

If higher turbine inlet temperatures are to be used, two approaches for upgrading hot-end component life are available: (1) the use of cooling processes, and (2) the development and use of materials with higher temperature capability. The cooling approach is one of the less desirable methods, since it detracts from overall engine performance due to the use of compressor air. The second approach periodically has focused considerable attention on the refractory metals: columbium, molybdenum, tantalum, chromium, and tungsten. These metals possess mechanical properties generally acceptable for high temperature turbine engine usage, although their susceptibility to environmental effects (oxidation) generally imposes severe limitations on their utility in air breathing turbines. Of these refractory metals, the low density and more advanced alloy systems of columbium rank it as a very attractive candidate for potential turbine applications. To date, considerable effort has been expended by gas turbine manufacturers and refractory metal producers in an attempt to develop a columbium alloy capable of withstanding temperatures in excess of 2500°F. The principal disadvantage with columbium, however, is its poor oxidation resistance, which necessitates a self-healing protective coating system for satisfactory gas turbine engine operation.

As higher turbine inlet temperatures are developed in a gas turbine engine, the temperature effects are primarily imposed on the turbine vanes and blades in the exhaust stream. Although each of these hot-end

components must survive in a high temperature environment, there are different material characteristics each component requires for adequate service life.

As a prerequisite for the successful performance of turbine blade components, candidate columbium alloys must exhibit adequate creep and oxidation resistance to withstand the high rotational stresses and corrosive high temperature products of combustion encountered during engine operation. In addition, a turbine blade must possess both high and low cycle fatigue strength and be capable of withstanding foreign object damage.

A turbine vane, on the other hand, being a non-rotating component not subjected to high centrifugal forces and vibratory stress that could lead to high cycle fatigue failures, enjoys a less critical role than a blade. However, turbine vanes generally experience higher temperature and oxidation resistance and coatability are of prime importance. In addition, significant cyclic and steady state stresses exist in a vane as a result of severe radial and circumferential thermal gradients. To resist these stresses and the resultant buckling, it is necessary that a columbium alloy vane material exhibit good high temperature creep and stress rupture strength. Also, the thermal gradients coupled with extreme transients from hot starts and engine accelerations, result in cyclic stresses, and thermal fatigue can occur.

Because of the complex dynamic nature of a blade application which necessitates maximum strength and reliability, initial use of columbium-base alloys in experimental gas turbines has generally been oriented toward vane applications where the static nature of the component is less likely to result in catastrophic failure should structural distress be encountered. The severity of the oxidation problem has rendered past attempts unsuccessful at introducing columbium alloys in high temperature gas turbine vane applications. Recently, however, new interest has been revived in introducing columbium into advanced engine designs. Encouragement grows from improvements in protective coating systems culminating in an Air Force engine evaluation of wrought columbium alloy vanes and the reported development of alloy substrates exhibiting improved oxidation resistance.

In conjunction with recent improvements in protective coating systems and columbium alloy development, technology in the precision casting of reactive and refractory materials has also advanced, holding the promise for improved economy and design efficiency. In particular, recent product improvement work with investment cast titanium performed by Avco Lycoming¹ at the request of the Army Materials and Mechanics Research Center has demonstrated that complex configurations can be cast to finished dimensions with virtually no surface reaction with either the molding material or surface environment. The fact that extremely reactive, high melting point materials such as titanium alloys can be investment cast is significant, particularly when the concept is extrapolated to refractory materials which are known to share a common degree of casting complexity.

In view of the combined technological advances recently made with respect to protective coating systems for columbium alloys, oxidation-resistant columbium alloy development, and reactive metal casting techniques, thought has been advanced toward the casting of columbium into airfoil shapes. Successful implementation and marriage of these latest developments into gas turbine hot-section design could well result in a quantum jump forward in gas turbine capability and efficiency. It is the purpose of this program to determine the feasibility of producing investment cast columbium alloy vanes with oxidation and mechanical property potential consistent with United States Army/Avco Lycoming advanced engine requirements.

II. TECHNICAL APPROACH

A. PROGRAM PLAN

The program to establish the feasibility of casting columbium alloys consists of two phases. In Phase I, casting mold and mold preheat temperature variations will be investigated and optimized. Utilizing these optimized parameters, stress rupture and thermal fatigue test bars and turbine nozzle vanes will be produced for mechanical property and oxidation thermal fatigue evaluation in Phase II.

¹U.S. Army/Avco Lycoming Product Support and Component Improvement Program CY 1968/69 Quarterly and Final Report Nos. 3162.1/2/3/4 (Improved Compressor) and 4212.2/3 (Investment Cast Titanium Impeller)

Subcontractor for the Phase I casting activity will be the REM Metals Corporation of Albany, Oregon. Basis for the selection of REM in this effort has been the successful performance of their patented REMET molding technique in the casting of titanium. Although REM has had no previous experience with the casting of columbium, titanium's highly reactive nature and extreme melting temperature are similar to those requirements for columbium. As a result, it was considered that the REM molding process offered excellent potential for the precision casting of columbium.

Basically, the REMET mold making technique is a modification of the shell molding process. The important feature which eliminates or minimizes surface contamination, is the use of metallic tungsten layers at the mold cavity casting interface. To produce a mold, a wax pattern of the desired shape is dipped into a tungsten oxide powder slurry to form a first layer. Dry powder is then applied to the wet layer. This process is repeated until the desired number of layers is built up. A backup layer of stabilized zirconia is added as a final step. Following removal of the wax by a chlorinated solvent, and baking at 600°F to drive off moisture, the molds are completed by reducing in hydrogen at 2150°F and curing in vacuum at 2400°F.

The selection of two columbium alloys for inclusion in the casting program was based on the five following factors:

1. Composition
2. Availability
3. Cost
4. Coatability
5. Mechanical properties

To insure that the spectrum of columbium castability would be covered, two alloys with widely differing compositions were desired. Based on the above requirements, columbium alloys B-66 and WC-3015 were selected for this program. The great difference in the compositions of the two alloys is shown in Table I. Westinghouse Corporation's B-66 alloy has been a candidate alloy for turbine vane application in a number of experimental programs conducted by governmental agencies and industrial organizations. Published results² indicate excellent coat-

² Hauser, H.A. and Holloway, J.F., Jr., Evaluation and Improvement of Coatings for Columbium Alloy Gas Turbine Engine Components, Technical Report AFML-TR-66-186, Part II, Pratt & Whitney Aircraft Corporation, May 1968.

TABLE I
NOMINAL ALLOY COMPOSITION (WEIGHT PERCENT)

<u>ELEMENT</u>	<u>ALLOY</u>	
	<u>WC-3015</u>	<u>B-66</u>
Hafnium	29.0	-
Tungsten	16.0	-
Tantalum	1.0	-
Carbon	0.1	-
Zirconium	1.5	1.0
Vanadium	-	5.0
Molybdenum	-	5.0
Columbium	Balance	Balance

ability and thermal fatigue life. Columbium alloy WC-3015, from Wah Chang Corporation, is a relatively new alloy designed for improved oxidation resistance. This is accomplished by high hafnium content (approximately 30%) of the alloy. Wah Chang data³ indicate elevated temperature stress rupture life to be superior to other uncoated alloys.

The first phase of the program, to determine the feasibility of producing precision columbium castings, includes a minimum of seventeen pours for each of the two alloys, B-66 and WC-3015. Initially, the fluidity specimen mold, prepared from the wax pattern depicted in Figure 1, will be used to determine both the optimum number of tungsten layers and also the optimum mold preheat temperature (in the photograph, the thin channels extending from the down-sprue are about 0.030 and 0.060 inch thick by one inch high).

Before the program pours commence, preparation of skulls for each of the two alloys in the tungsten layer evaluation will be completed. Skulls are necessary in the melting of columbium as they protect the copper crucible from damage during arc melting.

To prepare a skull for the melting of columbium, a vacuum consumable electrode arc melting furnace (Figure 2) is employed. To initiate melting, an arc is struck between the columbium alloy electrode and a slab of the same material in the water-cooled copper crucible. Following the initial pour, a cup or skull of the solidified alloy remains in the copper crucible. Different rates of expansion allow the skull to be removed from the crucible and saved until another melt of the same alloy is desired. For subsequent pours, the skull is reinserted into the copper crucible. After preparation of the skulls, the tungsten layer and mold preheat optimization phase of the casting program will begin. This involves the pouring of two fluidity molds for each alloy.

Initially, the effect of tungsten layers on cast surface quality will be investigated. The first four pours will be into three-layer tungsten molds with no mold preheat. The presence or absence of contamination on the cast columbium surface will determine whether a greater or fewer number of tungsten layers is necessary. A micro-hardness traverse

³ Wah Chang Albany Corporation, Technical Product Bulletin, WC-3015.

and metallographic examination of appropriate cross sections, and visual inspection will form the basis for this decision. Another four pours (two for each alloy) will then be cast into either two or four layer molds. Similarly, the third group of pours will be into either one or five -layer tungsten molds.

The next twelve pours utilizing the optimum number of layers determined by the above cold mold approach will then be directed at achieving the optimum mold preheat temperature. Three temperatures will be investigated using two pours of each of the two alloys, WC-3015 and B-66. The initial four pours will be 1000°F. Both visual and X-ray inspection of the fluidity castings will determine whether a mold preheat temperature of 500°F or 1500°F will be used for the next four pours. The last four pours in the series will use a preheat temperature of either less than 500°F or more than 1500°F, depending upon preceding results.

Phase I will be completed with the pouring of both alloys in the following mold configuration using optimum preheat temperatures and optimum number of tungsten layers:

1. Smooth stress rupture bars
2. Round thermal fatigue bars
3. Thermal fatigue paddles
4. T55 first stage nozzle vanes

Presented in Figures 3 and 4 are schematic drawings of the above test bars. Shown in Figure 5 are photographs of a thermal fatigue paddle and a first stage T55 vane.

Phase II of the program will utilize selected castings, as produced above, for the evaluation of mechanical properties. These parts will be coated with an oxidation resistant coating to be decided at the completion of Phase I. The tests will consist of:

1. Oxidation -thermal fatigue testing (paddles or round bar specimens). Paddles are preferred for testing in the Avco Lycoming experimental test rig (Figure 6). However, should unresolvable castability problems be encountered, the testing shall be conducted on what should be a more castable round test bar specimen. Testing will consist of up to 5000 cycles in a natural gas fired rig to evaluate the coated alloys' potential for turbine vane applications. During the test, the specimens will be heated to 2100°F in 40 seconds, held at temperature for one and one-half minutes,

and then cycled out of the flame into a combination water jet and mist spray in 30 seconds, following which the cycle is repeated. Lycoming 701 coated nickel base alloys M3608, TRW VI A, and AF2-1DA will be utilized for baseline information.

2. Stress rupture (smooth specimen) in air at 2200°F at a stress that will cause failure in 100 to 300 hours.

Contingent upon the development of sufficient potential, turbine vanes representative of the most promising coating/alloy combination will be considered for T55 engine testing under high temperature operating conditions. Since engine evaluation of vanes is not a funded part of the present program, any decision to initiate such an effort will require a review and agreement by appropriate Avco Lycoming engineering and Army Materials and Mechanics Research Center personnel. The complete program is outlined in Figure 7.

B. QUARTERLY ACTIVITY

1. Material Procurement

Columbium alloy suitable for use as the consumable electrodes, and wax patterns for the previously described molds, were obtained during the first month of the program. The B-66 alloy was supplied to REM Metals by the Fansteel Metallurgical Corporation. The WC-3015 alloy was supplied by its developer, Wah Chang Corporation. A quantity of 250 pounds of material was obtained for the 17 pours (12.5 pounds) of each alloy, including material for the electrode stub, skull, and starting chips. Avco Lycoming supplied REM Metals with appropriate wax patterns of the T55 first stage nozzle vanes and wax patterns of special test specimens. The complete list of materials is described below.

- a. B-66 Alloy - 250 pounds
- b. WC-3015 Alloy - 250 pounds
- c. Thermal Fatigue Test Paddle Wax Patterns - 50 pieces

- d. Vane Wax Patterns, with Cores - 10 pieces
- e. Vane Wax Patterns, Solid - 15 pieces
- f. Preformed Ceramic Vane Cores - 6 pieces

2. Preliminary Columbium Casting Activity

Although not part of the original program, REM Metals undertook a preliminary effort to insure that their melting facility and REMET mold system did not contain major deficiencies which might not have been anticipated during the initial program design. Specifically, it was felt that this additional activity was required as columbium alloys had never been investment cast before. Because of this, basic columbium melting and casting information which might have been applicable to this program was unavailable. Consequently, preliminary activity was initiated with the Cb-752 alloy, the election of which was based on availability and cost. Accordingly, two attempts were made to vacuum consumable electrode melt the Cb-752 alloy and produce a usable skull. After a successful skull had been made, two different four-bar test bar clusters were cast and were metallurgically examined to obtain preliminary information regarding the compatibility of the REMET mold system with the Cb-752 alloy.

Subsequent to the activity with the Cb-752 alloy, skulls were produced for both the B-66 and WC-3015 alloys and two four-bar clusters of test bars utilizing three tungsten layer molds and no mold preheat were investment cast. Following casting, the B-66 and WC-3015 test bars were sectioned and examined metallurgically to determine the presence of any mold-metal reaction which might have occurred in the mold upon solidification from the molten state.

3. Program Pours

Three layer tungsten fluidity molds were prepared for the

initial B-66 and WC-3015 program pours. A total of two cold (no mold preheat) fluidity molds were poured for each alloy. Program status at the end of this reporting period was approximately six weeks behind schedule due to unexpected delays which were encountered during a major plant move on the part of REM Metals Corporation, in which their entire facilities were relocated. It is anticipated that lost time will be regained through accelerated efforts and the REM portion of the program will be completed on schedule.

III. DISCUSSION OF RESULTS

A. PRELIMINARY COLUMBIUM CASTING ACTIVITY

A peculiarity of the columbium alloys investigated is the shape formed at the arc end of the electrode during melting. As shown in Figure 8, the columbium alloys, when melted, exhibited an electrode geometry ranging from relatively flat to concave upward. The Cb-752 alloy exhibited the most pronounced concave geometry. Because of this, the arc wandered about the periphery of the electrode seeking the point closest to the melting crucible. As a result, an inherent instability in arc voltages was created, causing a power fluctuation that was difficult to control. In terms of melting difficulty, the Cb-752 material required two tries to obtain a good skull, whereas the smoother melting B-66 and WC-3015 alloys were melted on first attempts. On the first Cb-752 melt, the difficulty in controlling the arc caused perforation of the copper crucible and admission of water into the system. A second attempt produced a successful 25-pound melt from which a skull was obtained. Relative to test bar molds, the first cluster of Cb-752 test bar molds were poured with good fill. However, severe overheating of the copper crucible occurred due to an inadequate cooling water flow rate (75 gallons per minute). The second test bar pour, which also yielded good fill, was made with an increased flow rate (95 gallons per minute) with no overheating difficulty. The Cb-752 electrode stub, skull, and test bar mold are shown in Figure 9. A closeup view of the fill obtained on the Cb-752 test bars is presented in Figure 10. As for the B-66 and WC-3015 test bars, very good fill was obtained (Figure 11) demonstrating that relatively complex shapes can be cast with columbium alloys.

Microstructural examination of the test bars representative of the Cb-752 and WC-3015 alloys showed no evidence of surface contamination from a mold-metal reaction. However, as shown in Figure 12, the B-66 alloy revealed evidence of surface contamination ranging, in some areas, up to 0.008 inch. Grain size in the Cb-752 and WC-3015 castings was a fine and uniform ASTM 6 as opposed to a slightly less uniform ASTM 4 to 6 structure evidenced in the B-66 material. Figure 13 depicts a typical microstructure comparison in investment cast Cb-752, B-66, and WC-3015 alloys.

B. PROGRAM POURS

Visual inspection of the B-66 and WC-3015 cast fluidity specimens indicated poor fluidity, which was not surprising considering the fact that the molds were not preheated. By comparison, the WC-3015 alloy exhibited slightly better fluidity than B-66. A photograph shows the appearance of the B-66 and WC-3015 fluidity specimens in Figure 14.

Based on the satisfactory performance of the WC-3015 cold three-tungsten layer molds, the next two cold mold pours for this alloy will be made with two tungsten layers. Further casting of the B-66 alloy, however, will be performed with four tungsten layers in an attempt to eliminate surface contamination.

C. MOLD SYSTEM

Mold cracking became a problem during the mold preparation phase of both the preliminary and planned program activity. The cause of this cracking is apparently the result of thermal stresses built up during the extremely high temperature cycle to which the molds are exposed, combined with the difference in thermal expansion coefficients between the tungsten layers and the zirconia layer. Cracked molds were salvaged by externally reinforcing them with wire. Attempts are being made to correct the cracking problem by using slower heating and curing temperature cycles to reduce thermal gradients.

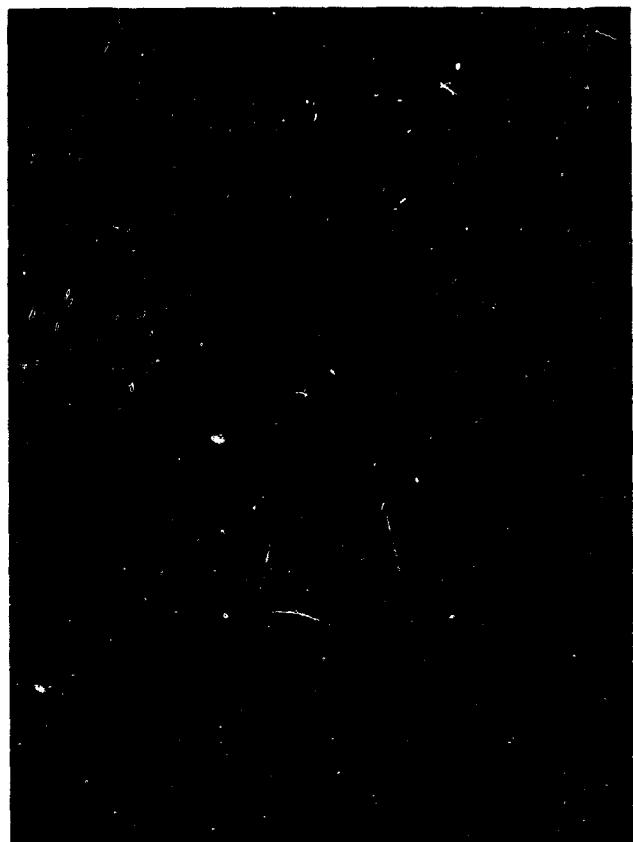


Figure 1. Typical Fluidity Specimen Wax Pattern.

¹³
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Figure 2. Skull Furnace Utilized to Cast Columbium Base Alloys.

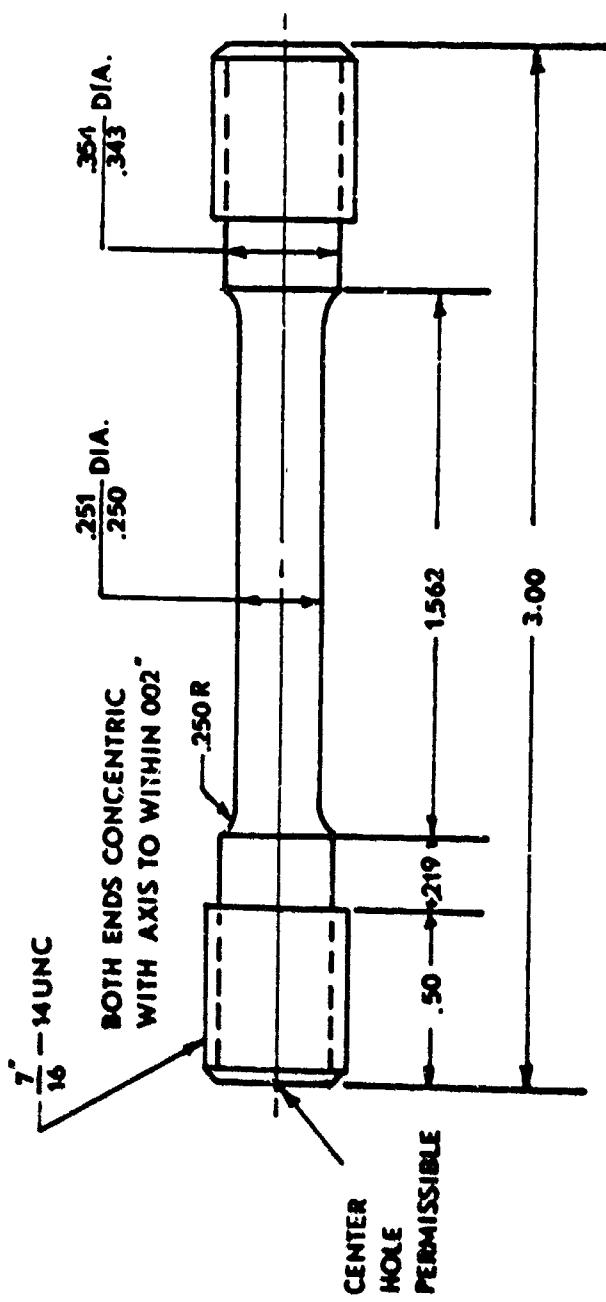


Figure 3. Smooth Stress Rupture Bar.

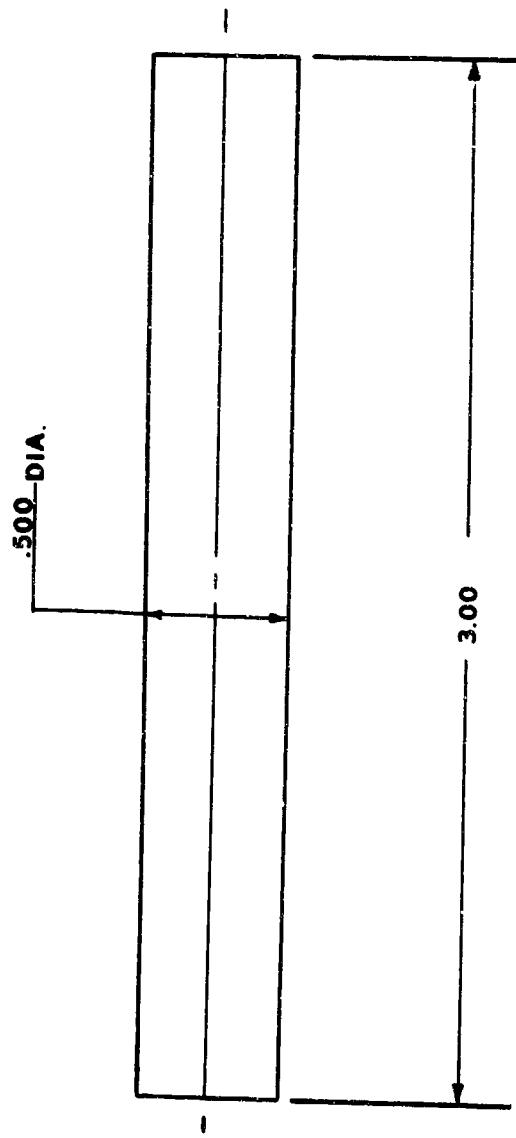


Figure 4. Round Thermal Fatigue Bar.

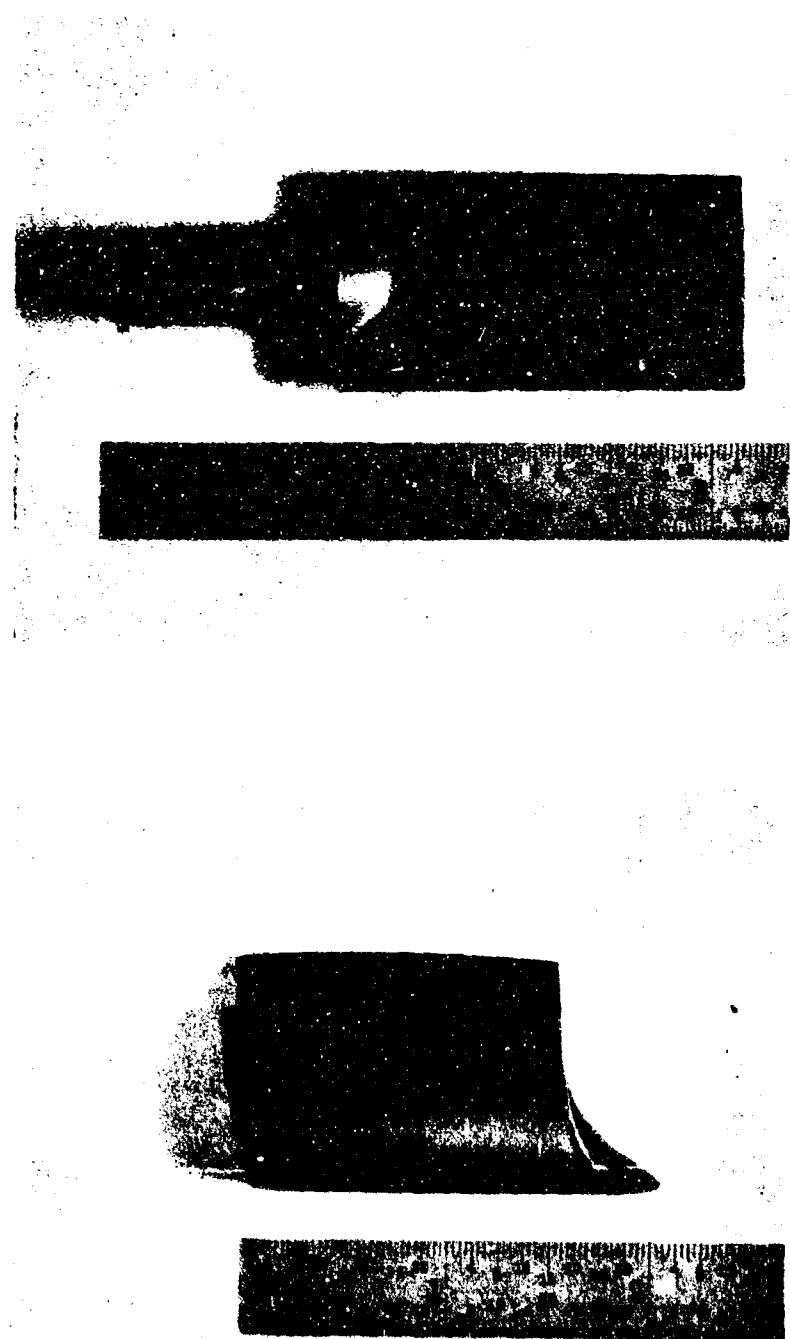


Figure 5. Thermal Fatigue Paddle (Top) and T55 First Stage Nozzle Vane.

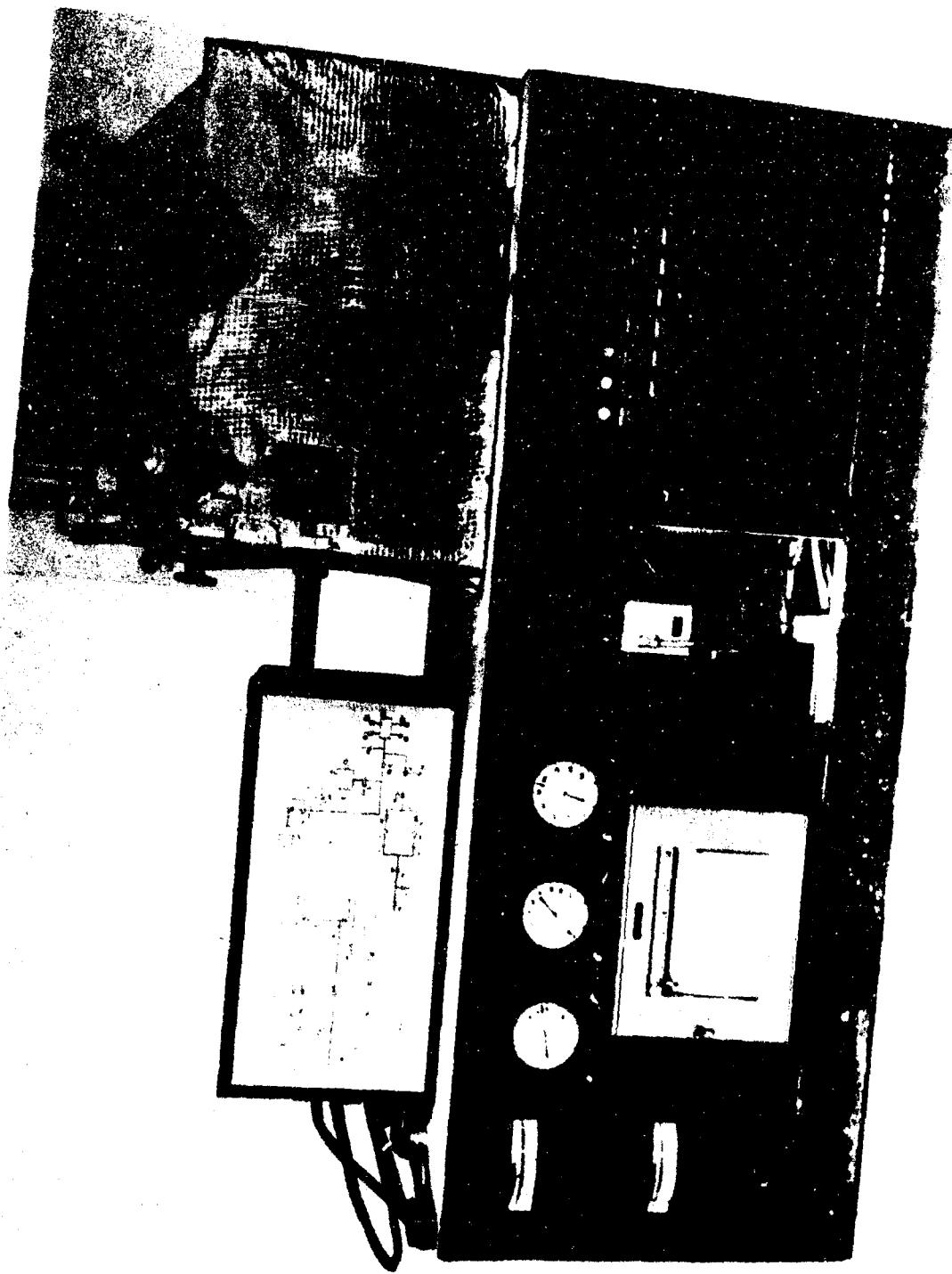


Figure 6. Avco Lycoming Oxidation - Thermal Fatigue Test Rig.

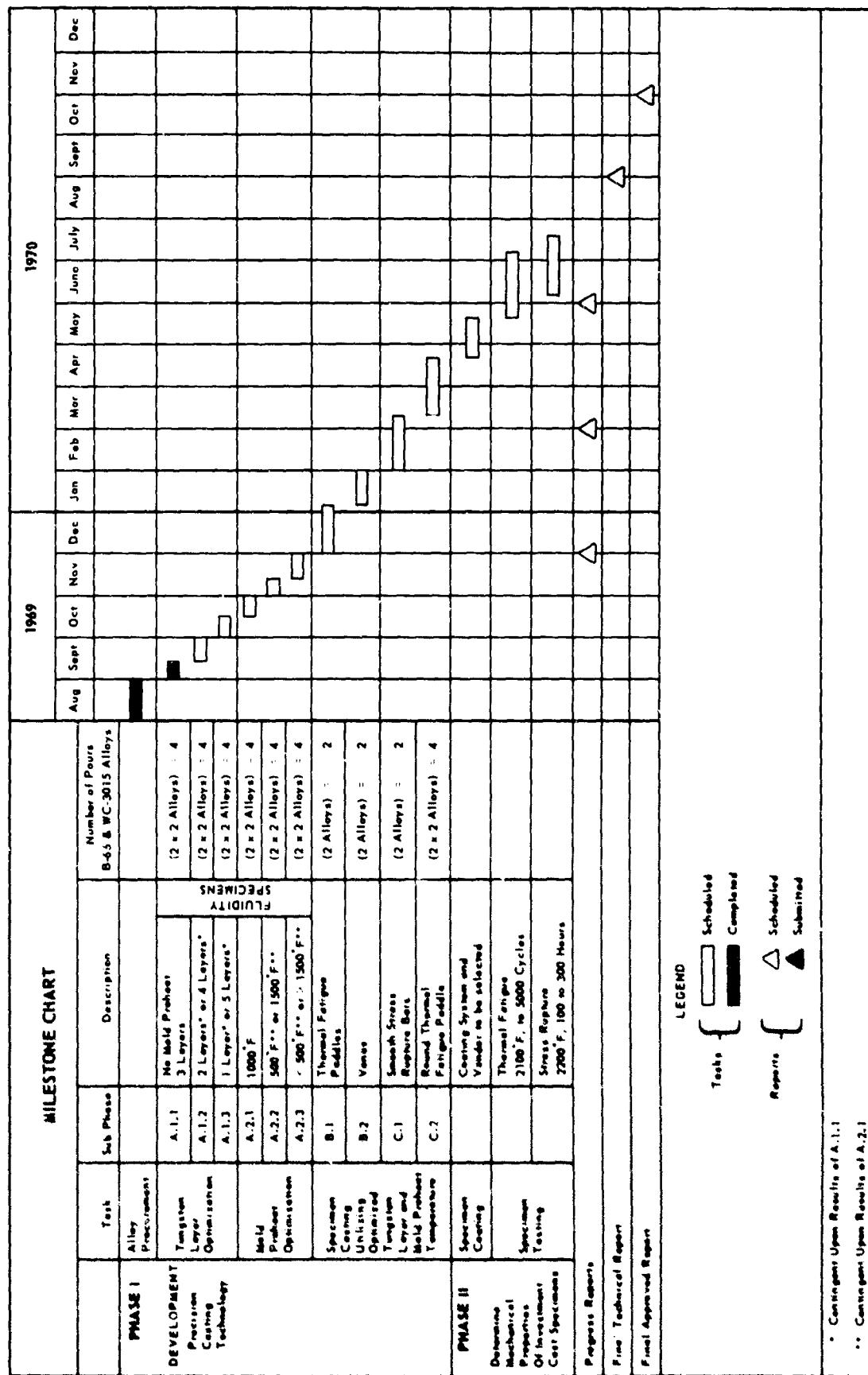
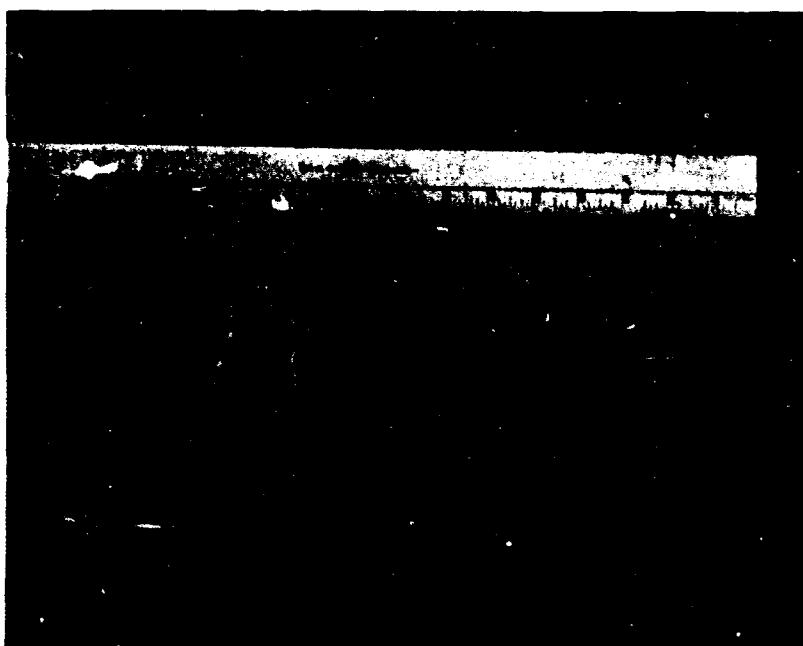


Figure 7. Cast Columbium Program.



Cb-752



WC-3015

Figure 8. Columbium Electrode Stubs.

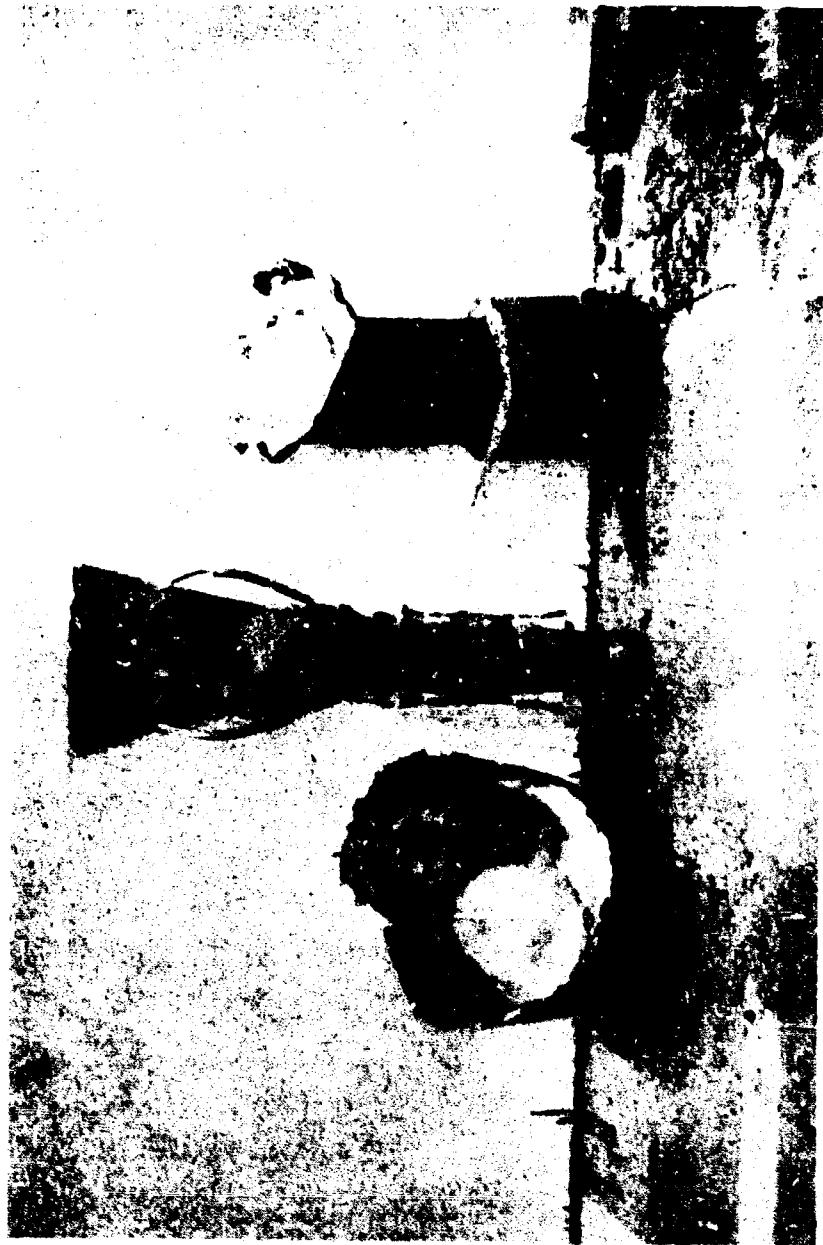


Figure 9. From Left to Right: Cb-752 Columbium Alloy Skull, Poured Test Bar Mold, and Consumable Electrode.



Figure 10. Cb-752 Precision Cast Test Bars Following Casting In A Three Tungsten Layer Cold REMET Mold (Mold Material Has Been Partially Chipped Away).

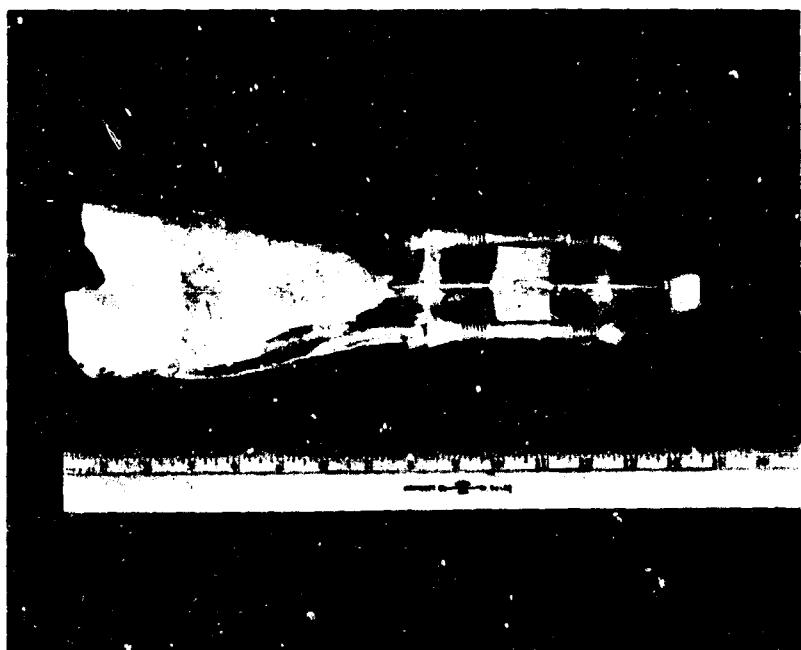


Figure 11. Typical Fill Developed for Cast B-66 Columbium Alloy Test Bars (Three Layer Tungsten Molds, No. Preheat).



**Etchant: 10 Lactic
10 HNO₃
1 HF**

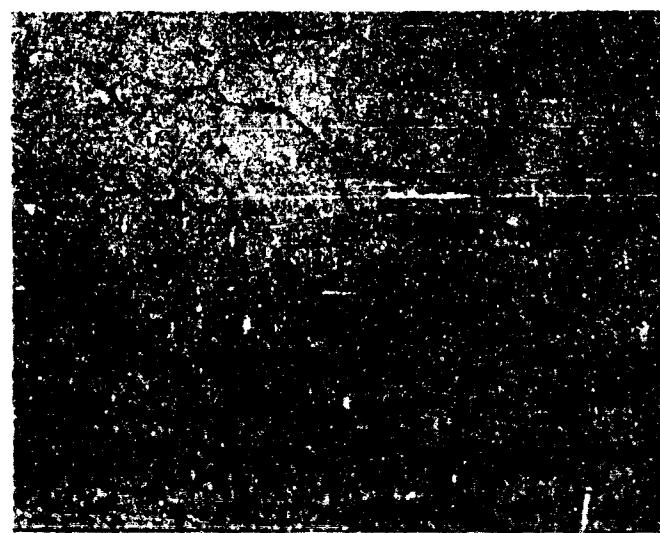
Mag: 100X

Figure 12. B-66 Surface Contamination.



Cb-752

15 Lactic
6 HNO₃
4 HF



B-66

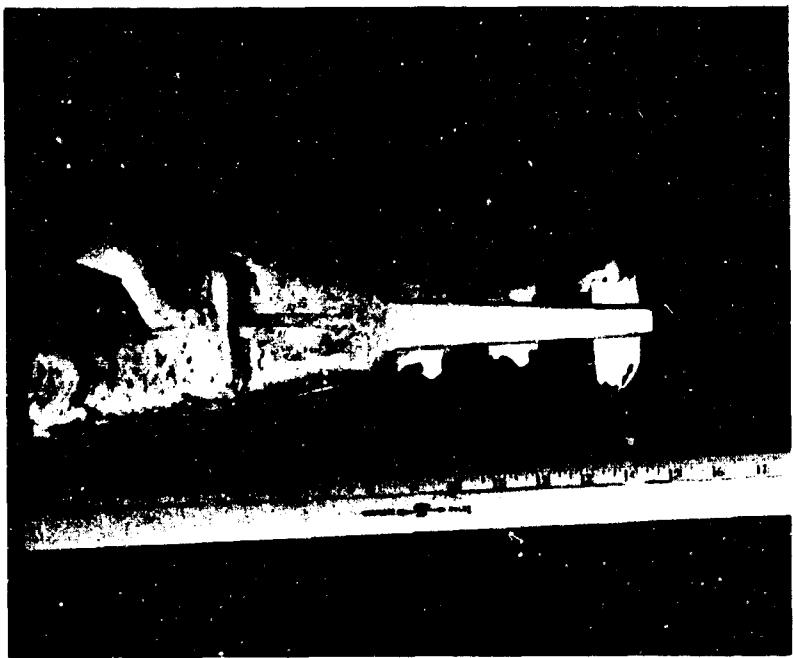
10 Lactic
10 HNO₃
1 HF



WC-3015

Etchants: Lactic - Phosphoric Acid
Anodize

Figure 13. Typical As-Cast Columbium Alloy Microstructures (Mag: 100X).



B-66



WC-3015

**Figure 14. Appearance of B-66 and WC-3015 Fluidity Specimens
(Three-Layer Tungsten Molds, No Preheat).**

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